

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant	:	Christopher Brett Ward
Serial Number	:	10/501,277
Filing Date	:	September 3, 2004
Title	:	Hydrometallurgical Processing of Manganese Containing Materials
Examiner	:	Arun S. Phasge
Group Art Unit	:	1795

DECLARATION OF MICHAEL J. NICOL UNDER 37 C.F.R. §1.132

I, Professor Michael J Nicol, of Mount Pleasant, Western Australia, Australia, do declare and state as follows:-

Personal Background

1. (a) I retired at the end of 2009 as Professor and Chair of the Department of Extractive Metallurgy at Murdoch University, located in Perth, Australia, and in 2007 as the Program Manager, Leaching, Separation and Reduction, at the AJ Parker Cooperative Research Centre for Hydrometallurgy, also located in Perth, Australia.

(b) I declare that the following facts, matters and instances are within my own knowledge and belief unless otherwise indicated to the contrary herein.
2. A copy of my *curriculum vitae* is annexed hereto as Annexure MJN-1.
3. I have particular experience and expertise with regard to hydrometallurgy, in particular leaching, recovery and electrochemical processing of base metals and precious metals, and I have specific and relevant experience in research and development relating to manganese chemistry and processing.
4. As of the filing date of the above-identified U.S. Patent Application (hereinafter "the Application"), and also as of the priority date to which the Application is entitled, being 10 October 2002 (hereinafter "the Priority Date"), I possessed a generally similar level of experience and expertise. The following comments and observations are provided on the basis of my knowledge as at the Priority Date unless otherwise

indicated.

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5. I have read and understood the patent specification filed in respect of the Application, including the claims thereof as most recently proposed to be amended, and a copy of this specification is attached hereto as Annexure MJN-2.
6. I have read and understood the four Office Actions that have issued in respect of the examination of the Application, copies of which are attached as Annexure MJN-3.

Claim Rejection – 35 USC § 112

7. I understand that the Examiner believes that the new limitations introduced into the claims in the most recently amended claims, as per the content of Annexure MJN-2, were not described in the specification in such a way as to reasonably convey to one skilled in the relevant art that the inventor, at the time the application was filed, had possession of the claimed invention.
8. However, it is my view that the description set out at page 1, lines 18 to 27, and page 2, lines 8 to 26, clearly shows that the process of the invention is able to be distinguished from the prior art on the basis that “no sintering or roasting pre-treatment step of the feedstock is undertaken”. I refer particularly to the following sentences:

"Low grade manganese dioxide feedstock (<40% Mn) are presently uneconomic to process using conventional roast-reduction and sulphuric acid leaching to produce manganese sulphate. High grade ores (>40% Mn) are needed to justify the economics of the roast reduction process" (page 1, lines 18 to 21)."

and

"Further, the use of a hydrometallurgical route for the reduction of Mn(IV) negates the need for the use of gas fired kilns or fluid bed reactors, feed stocks no longer need to be heated to about 1000°C and then cooled prior to leaching, and there is lesser need for carbon input, which in turn results in lower greenhouse gas emissions" (page 2, lines 12 to 16).

9. These passages clearly would convey to one of ordinary skill in the art that the previous art to process MnO₂ ores needed to have a roasting step (read sintering step) and needed to use relatively high grade ores (as confirmed in the Kasaaian patent), unless non Mn(IV) manganese ores, such as Mn₃O₄ ores, are processed.

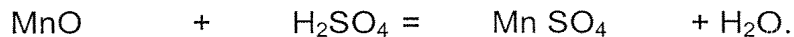
Claim Rejection – 35 USC § 103

10. I have read and understood each of US Patent 5932086 (“Kasaaian”) and US Patent 3301777 (“Leonard”) which I understand form the basis for the Examiner’s rejection of the most recently amended claims of the Application for obviousness.
11. As both previous patents cited by the Examiner are predominantly related to electrolytic manganese metal (EMM) technologies, it is now prudent to give some background to the differences between the two electrowinning technologies and practices, to show why certain points raised by the Examiner are irrelevant and should not be used as a basis for rejecting the claims. It is also my opinion that one of ordinary skill in the art would not combine the Kasaaian and Leonard patents to produce the invention described and claimed in the Application, as neither of those patents deals with the fundamental purpose of the invention, which is: being able to prevent or minimize the formation of lower valence oxy-sulfur containing species such as dithionate, which are known to occur during the reduction of higher oxides of manganese using sulfur dioxide, and are a major process inefficiency, resulting in the formation of different products as contaminants, thus discouraging this route from being utilised in the industry previously.
12. While manganese metal is plated from an alkaline manganese solution onto stainless steel cathode sheets in the presence of ammonium ions at relatively high current densities ($\sim 200 \text{ Am}^{-2}$) it is general practice to add a low level of reducible sulfur containing species such as SO_2 , dithionate, xanthate, sulfide or sulfites in the catholyte to suppress the hydrogen ion reduction reaction which would form hydrogen gas. It is also important to note that the use of SO_2 and other reducible sulfur species has become more commonplace and at higher levels in recent years as the EMM industry moves away from the addition of selenium dioxide in their production facilities to minimize the negative product and toxic effects on workers of selenium being present.
13. The Leonard patent states that “this invention relates to the production of electrolytic manganese metal. More specifically, this invention relates to the use and construction of anodes in the electrolytic production of manganese metal”. As this patent is promoting the merits of its new design and construction of titanium anodes to overcome the corrosion by the reductive removal of the TiO_2 passivating layer and potential failure of titanium anodes at the solution-air interface, the patent must show that the design and construction of the anodes must be able to operate successfully under all conditions expected within an operating EMM cell. Thus,

Leonard has rightly pointed out that his invention will not be affected by any of the potential conventional additives that might be included in the electrolyte, such as reducible sulfur compounds (column 4, line 29), and more specifically, "between about 0.05 and 1.0 grams per litre of reducible sulfur compounds such as dithionates, xanthate, sulfides, sulfides or sulfur dioxide" (column 4, lines 39+). When these additives are used, it is generally accepted that some of the compounds will find their way into the anolyte as well.

14. If these additives, or others, are not added to the catholyte, then the current efficiency of the plating process can potentially drop to below 70%, which will severely affect the economics of the EMM process. When additives are utilised, on the other hand, much higher current efficiencies are expected as the hydrogen gas formation reaction is minimized in favour of the metal plating reaction.
15. The Kasaaian patent again essentially relates to electrolytic manganese metal (EMM) technology and only briefly mentions the production of electrolytic manganese dioxide (EMD), but importantly Kasaaian does not give any reference to examples of using his method for producing EMD. Kasaaian never mentions that his proposed invention can be utilized in a continuous recirculating hydrometallurgical process where spent acid or manganese-depleted return electrolyte solutions get recycled back to the start of the initial leach step. While this might be considered as obvious in engineering a hydrometallurgical process, it is highly likely that Kasaaian did not use return electrolyte solutions at all, or for a number of cycles, and therefore did not encounter the steady build-up of unacceptable levels of manganese dithionate in the leach solutions as more and more dithionate is formed during each successive cycle. As said, without doing this "lock-cycle" testing in the EMD process and testing for dithionate levels and their effects on the products, Kasaaian may not have been aware that his technology could not be utilized for commercial EMD production.
16. Kasaaian's lack of by-product dithionate discussion can also be attributed to the fact that he only considered that his proposed process was applicable to manganomanganic oxides (Mn_3O_4), which have one third of the manganese units in the $Mn(4+)$ state as compared with MnO_2 which has 100% of its manganese units in the $Mn(4+)$ state. Thus, to achieve a manganese sulfate solution from a reductive leach of manganomanganic materials, only one third of the manganese needs to undergo a reduction reaction with sulfur dioxide as the other 66% of the manganese units will simply be solubilised by the exothermic dissolution reaction of MnO with

sulfuric acid.



17. Thus the subject invention deals with a 3 fold more intensive reductive leaching situation than does the Kasaaian patent, and therefore Kasaaian's levels of dithionate by-product formation would be expected to be only one third of what Kasaaian would have seen had he utilized a 100% MnO_2 containing ore (which does not consume sulfuric acid). By not performing a number of leach cycles using return electrolyte, and only doing a reductive leach at one third the intensity, the chances of Kasaaian observing and appreciating the negative effects and inefficiencies of dithionate formation may have been very limited. Thus, again I stress that the lack of appreciation of the formation of dithionates and methods for controlling its formation by Kasaaian is a key difference, showing that the Application discloses and claims a technological advance over the prior art, and it is therefore my opinion that this fact alone should be sufficient basis to confirm that the Application clearly represents a significant patentable process improvement.
18. Neither of the Kasaaian or Leonard patents should prevent the patentability of the claims of the Application, as neither cited patent deals with the invention that is disclosed in the Application. The subject invention controls (read minimizes to trace amounts) the amount of by-product dithionate (in no way purposely added as an additive) that is generated in a continuous recirculating sulfur dioxide leach. This invention then allows this method of solubilising MnO_2 containing ores to be linked to an electrowinning tankhouse to enable the safer (i.e. with greatly reduced H_2S emissions) and higher quality (avoids extra contaminant sulfur species being incorporated in the EMD) production of products to suit the high quality alkaline battery market.
19. Claim 29 of the Application clearly mentions a manganese dioxide feedstock, which had previously been described as being a Mn(IV) containing ore (generally considered in the industry to be MnO_2); the latter is a pyrolusite or cryptomelane crystal type mineral containing natural ore. This is demonstrably different than the mangomanganic natural ore discussed in Kasaaian, which is described as Mn_3O_4 and which is a hausmannite crystal containing ore of lower valence. Mangomanganic material (Mn_3O_4) can also be produced by sintering or heating MnO_2 ores in a kiln or fluid bed reactor, but again, this heating or sintering step is made redundant by the invention of the Application. It is a key facet of the invention to thus be able to efficiently leach MnO_2 ores, whereas this would not be the case

with Kasaaian.

20. It is highly unlikely that Kasaaian even considered dithionate levels, due to the absence of any disclosure in Kasaaian of recirculating process steps being performed. Also, the significantly lower average manganese valence state in the manganomanganic ore required far less sulphur dioxide for reduction, resulting in less dithionate being formed. These comments are again very relevant to showing that claim 41 would not have been obvious to one of ordinary skill in the art. Consideration of claim 41 should thus not be influenced by the different natural or sintered feed stocks utilised in the Kasaaian patent or be affected by the addition of reduced sulfur containing additives to the EMM electrowinning step to overcome a current efficiency issue in the Leonard patent.
21. Accordingly, it is my strongly held opinion that the invention as described and claimed in the Application would not have been obvious to one of ordinary skill in the art at the Priority date, whether possessed of the Kasaaian and/or Leonard patents or not, due in large part to the differences in manganese containing ores the subject of the respective processes, the specific contemplation of a need to maintain low dithionate ion levels, and the express lack of a sintering or roasting pre-treatment step of the feedstock, each as explained above.

I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the Application or any patent issuing thereon.

DECLARED at CAPE TOWN in SOUTH AFRICA

this 22 day of FEBRUARY 2010.



Professor Michael J Nicol

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This is Annexure MJN-1 referred to in my Declaration made this 22 day
of FEBRUARY 2010.

CURRICULUM VITAE

Michael J Nicol

1. Personal Details

Current Position : Retired Professor of Extractive Metallurgy, Murdoch
University
Manger AMIRA P705B project "Electrowinning of Base Metals"

2. Education, Qualifications and Employment

Education and qualifications:

University of the Witwatersrand, Johannesburg, South Africa
BSc(Hons) 1961
PhD 1964

Brief employment history prior to current position at Murdoch University:

Postdoctoral Fellow and Assistant Professor

Texas A&M University 1964/65
Purdue University 1966/67

1968-1970
Lecturer; Dept of Chemistry, University of Natal, Durban, South Africa

1971-1994
Mintek, Johannesburg, South Africa

Group Leader Electrochemistry
Director: Mineral and Process Chemistry Division
Director: Hydrometallurgy Division
Chief Director

1994-1997
Mintek Research Professor: University of the Witwatersrand.

1997- 1999
Professor of Extractive Metallurgy, Murdoch University

1999-
Manager - Amira P705B project on base metal electrowinning
Consultant – BHPBilliton Heap Leach project
Research Advisor and Supervisor – Extractive Metallurgy, Murdoch University

3. Awards

Mintek Gold Medal (1982) for advances in uranium processing.
South African Institute of Mining and Metallurgy Gold Medal (1985)
TMS Distinguished Lecture Award at the TMS Conference, San Antonio, 1998.

4. Professional Societies

Fellow of the S. African Institute of Mining and Metallurgy.
Fellow of the Australasian Institute of Mining and Metallurgy
Member of the editorial board of the international journal “Hydrometallurgy”
(1975-1983)

5. Other Professional Activities

Member of the Evaluation Committee (Engineering) of the Foundation for Research Development in South Africa(1994-1997)

Member of Planning and Executive Committee of A J Parker Cooperative Research Centre for Hydrometallurgy (1997- 2006).

Manager for the Leaching, Separation and Reduction Programme in Parker Centre (1997- 2006).

Member of MRAC committee of Minerals and Energy Research Institute of Western Australia. (2003-2007)

6. Research, scholarship and professional activities

Research

The development of a world-class research activity in hydrometallurgy in collaboration with the A J Parker Cooperative Research Center for Hydrometallurgy has been the main focus of activities since joining Murdoch University in July 1997. Since that time, the research programme has expanded to include a number of PhD, Masters and Honours students. In addition, there are now 4 post-doctoral fellows working in the group.

Significant links with industry have been established by way of several ARC Linkage projects involving a number of industrial partners, participation in the AMIRA P420A and P420B Gold Technology programmes and the management of the international AMIRA P705 Base Metal Electrowinning project which involves companies from Australia, Chile, South Africa, Canada and USA. In addition to the A J Parker Centre, over 30 companies have recently or are presently directly contributing to the postgraduate research programme.

The main research interests are linked to the physicochemical basis for hydrometallurgical processes. Major contributions have been made in the application of electrochemistry to leaching, precipitation and metal winning in both precious and base metal processes. Other areas of research are the kinetics of ion exchange and solvent extraction processes and the digital simulation of heterogeneous processes.

In terms of research and development work on the hydrometallurgy of manganese, the following activities have been undertaken either directly or as the research director of the relevant work.

1. Fundamental studies of the kinetics and mechanisms of the processes involved in the electrowinning of manganese metal (EMM) and electrolytic manganese dioxide (EMD). This work was conducted over 5 years at Mintek in South Africa. As part of this research, a fully integrated pilot plant for the production of EMD was operated for several months using arc furnace dust from a ferromanganese operation.
2. Development of special ion-exchange resins for the recovery of manganese from plant effluent streams.
3. Operation of a pilot plant at Murdoch University for the production of EMD using the process which is the subject of this patent application. The development and operation of a method to assess the battery activity of the product EMD was also undertaken.
4. Small scale ion-exchange testwork to assess the technical feasibility of a resin-in-pulp process for the recovery of manganese from leach pulps.
5. Consulting activities for the MMC company in South Africa on the electrowinning of manganese metal.
6. Several visits to both EMM and EMD plants in both South Africa and Brazil.

Recent International Conference Presentations

Invited Plenary Lecture at the 4th International Symposium on Hydrometallurgy, Salt Lake City, August 1993.

Invited paper at International Conference on Leaching, Perth, Oct 1999.

Paper presented at the SME Conference, Salt Lake City, Mar 2000

Two papers presented at the TMS Conference, Nashville, Mar 2000

Invited paper at Minprex 2000 Conference, Melbourne, Sept 2000

Three papers presented at the TMS Conference, San Antonio, Mar 2001

Invited paper at International Conference on Separation Processes, Perth, Oct 2001

Invited paper at the TMS Conference, Seattle, Feb 2002

Three papers at the 5th International Conference on Hydrometallurgy, Vancouver, Sept 2003

Paper presented at Copper 2003, Santiago, Nov 2003

Paper presented at TMS Symposium on Arsenic, San Francisco, Feb 2005

Paper presented at SME Symposium in honour of Prof J D Miller, Salt Lake City, March 2005

Plenary paper at VII Meeting of the Southern Hemisphere on Mineral Technology, Ouro Preto, Brasil, Nov 2007.

Three papers presented at the TMS Conference on Hydrometallurgy in Phoenix, Sept 2008.

Paper presented at the Hydrometallurgy Conference in Antofagasta, Chile, May, 2009

Patents

J Preston and M J Nicol, Recovery of Rare Earth Oxides, S. Afr. Patent 882670, 1989

M.J. Nicol and G. Summer, The Measurement of Carbon Particles in Suspensions Thereof, S. Afr. Patent 823383, 1982

M.J. Nicol and G. Summer, Improvements relating to the Controlling of Processes, S. Afr. Patent 731213, 1973

M.J. Nicol, C.R.S. Needes and N.P. Finkelstein, Measuring Rates of Dissolution, S. Afr. Patent, 728807, 1972

M.J. Nicol and T. Tunley, Nickel Recovery from Laterite Ores, S. Afr. Patent, 958374, 1995

Honguang Zhang and M J Nicol, Thiosulfate process for the Leaching of Gold, Australian patent, 2005

M J Nicol, P Basson and E Muller, Chloride Heap Leaching of

Chalcopyrite, Chilean Patent, No 1355-07, 2007

M J Nicol, P Basson and E Muller, Chloride Tank Leaching of Chalcopyrite, Chilean Patent, No 1356-07, 2007

Referred Papers

68 papers prior to 2000

Since 2000

Nicol, M.J. and Guresin, N, "A Novel Approach to the Oxidation of Arsenical Refractory Gold Concentrates", *Minor Elements 2000*, C. Young(ed), SME, Littleton, Colorado, March 2000

Nicol, M.J. "The Monitoring of the Speciation of Iridium in the Separation of Iridium from Rhodium", *Iridium*, E.K. Ohrliner, R.D. Lanam, P. Panfilov, H.Harada(ed), TMS, Warrendale, Pennsylvania, pp351-359, March 2000

Nicol, M.J. and Kittelty, D., "The Electrocrystallisation of Nickel and its Relationship to the Physical Properties of the Metal." *Electrochemistry in Mineral and Metal Processing*, R. Woods, F.M. Doyle, S. Licht. (ed), The Electrochemical Society, Toronto, Canada, pp 362-373, May 2000

Lazaro, I and Nicol, M.J. "Some aspects of the electrochemistry of chalcopyrite under typical leaching conditions", *Electrochemistry in Mineral and Metal Processing*, R. Woods, F.M. Doyle, S. Licht. (ed), The Electrochemical Society, Toronto, Canada, pp 217-230, May 2000

Nicol, M.J. "Hydrometallurgy into the Next Millenium", *International Congress on Mineral Processing and Extractive Metallurgy*, Australasian Institute of Mining and Metallurgy, Publication Series No 5/2000, pp 11-16 September 2000.

Hindmarsh, K and Nicol, M.J. "A Novel Method for the On-line Analysis of Active Cyanide During the Cyanidation of Gold", *Cyanide: Social, Industrial and Economic Aspects*, C. Young, L. Twidwell and C. Anderson(ed), TMS, Warrendale, Pennsylvania pp151-162 February 2001

Ritchie, I., Nicol, M.J. and Staunton, W.P. "Are There Realistic Alternatives to Cyanide as a Lixiviant for Gold at the Present Time?", *Cyanide: Social, Industrial and Economic Aspects*, C. Young, L. Twidwell and C. Anderson(ed), TMS, Warrendale, Pennsylvania pp427-440, February 2001

Nicol, M.J. and O'Malley, G.P. "Recovery of Gold from Thiosulfate Solutions and Pulps with Ion-Exchange Resins", *Cyanide: Social, Industrial and Economic Aspects*, C. Young, L. Twidwell and C. Anderson(ed), TMS, Warrendale, Pennsylvania, pp469-484, February 2001

Nicol, M.J. and Kittelty, D, "The Electrocrystallisation of Nickel and its Relationship to the Physical Properties of the Metal." *Electrometallurgy 2001* , J A Gonzalez, J E Dutrizac, G H Kelsall (ed), Canadian Institute of Mining, Metallurgy and Petroleum, Toronto, August 2001

Nicol, M.J. and Lazaro, I, "The role of Eh measurements in the interpretation of the kinetics and mechanisms of the oxidation and leaching of sulfide minerals." *Hydrometallurgy*, vol 63, pp 15-22, 2002.

Nicol, M. J., "Developments in the Teaching of Hydrometallurgy in Australia.", *EPD Congress 2002*, P R Taylor (Ed), TMS, Warrendale, Pennsylvania, pp773-784, February 2002

Qing Liu, J. and Nicol, M.J., "Thermodynamics and Kinetics of the Dissolution of Gold under Pressure Oxidation Conditions in the Presence of Chloride", *Can. Met. Quart.*, vol 41, pp409-415 , 2002

Nicol, M.J. and O'Malley, G. "Recovering Gold from Thiosulfate Leach Pulps by Ion Exchange", *J.Metals*, vol 54, pp 44-46, 2002

Liu, J.Q. and Nicol, M.J., "Thermodynamics and Kinetics of the Dissolution of Gold under Pressure Oxidation Conditions in the Presence of Chloride", *Chloride Metallurgy 2002*, E. Peek, G van Weert (ed), Canadian Institute of Mining, Metallurgy and Petroleum, Montreal, pp 41-54, October 2002

M A Buswell and M J Nicol, "Some Aspects of the Electrochemistry of the Flotation of Pyrrhotite", *Journal of Applied Electrochemistry*, vol 32, pp 301-309, 2002.

S. Zhang and M J Nicol, "An Electrochemical Study of the Dissolution of Gold in Thiosulfate Solutions. I Alkaline Solutions", *Journal of Applied Electrochemistry*, vol 33, pp 767-775, 2003

M J Nicol and Z Zainol, "The Development of a Resin-in-Pulp Process for the Recovery of Nickel and Cobalt from Laterite Leach Slurries", *Int. J. Miner. Process*, vol 72, pp 407-415, 2003.

M J Nicol and N Guresin, "The Anodic Behaviour of Arsenopyrite and the Cathodic Reduction of Ferrate(VI) and Oxygen in Alkaline Solutions", *Journal of Applied Electrochemistry*, vol 33, pp 1017-1024, 2003.

I Lazaro and M J Nicol, "The Mechanism of the Dissolution and Passivation of Chalcopyrite – An Electrochemical Study", *Hydrometallurgy 2003*, The Minerals, Metals and Materials Society, Warrendale, Pennsylvania, vol 1, pp 405-418, 2003

D Kittelty and M J Nicol, "The Effects of Solution Impurities on the Properties of Nickel Cathodes", *Hydrometallurgy 2003*, The Minerals, Metals and Materials Society, Warrendale, Pennsylvania, vol 2, pp 1205-1218, 2003

M J Nicol and J.Q. Liu, "The Effect of Chloride Ions on the Oxidation of Pyrite Under Pressure Oxidation Conditions", *Hydrometallurgy 2003*, The Minerals, Metals and Materials Society, Warrendale, Pennsylvania, vol 1, pp 591-602, 2003

G. Senanayake, W.N.Perera and M J Nicol, "Thermodynamic Studies of the Gold(III)/(I)/0 Redox System in Ammonia-Thiosulfate Solutions at 25°C.", *Hydrometallurgy 2003*, The Minerals, Metals and Materials Society, Warrendale, Pennsylvania, vol 1, pp 155-168, 2003

M J Nicol and I Lazaro, "The Role of Non-Oxidative Processes in the leaching of Chalcopyrite", *Copper 2003*, The Canadian Institute of Mining, Metallurgy and Petroleum, Montreal, Quebec, Book 1, pp 383-394, 2003

A N Nikoloski, M J Nicol and J E Fittock "The Passivation of Iron during the Leaching of Pre-reduced Laterite Ores in Ammoniacal Solutions", *Electrochemistry in Mineral and Metal Processing VI*, The Electrochemical Society, Pennington, NJ 2003.

M J Nicol, A N Nikoloski and J E Fittock, "A fundamental study of the leaching reactions involved in the Caron Process", *International Laterite Nickel Symposium, TMS, Warrendale PA, 2004, pp369-384*.

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S. Zhang and M J Nicol, "The simultaneous oxidation of sulfide minerals and the dissolution of gold", *Arsenic Metallurgy* (G. Reddy and V. Ramachandran, ed) , The Minerals, Metals and Materials Society, Warrendale, Pennsylvania, 2005, pp 265-282

M J Nicol and H Miki, "Applications of the electrochemistry of fine mineral sulfides", *Innovations in Natural Resource Processing*, (C.Young et al ed.), SME, Littleton, CO, 2005, pp 179-192.

M J Nicol, "Developments in the Teaching of Hydrometallurgy in

Australia”, *Hydrometallurgy*, vol 79, pp 23-30, 2005

W M Perera, G Senanayake and M J Nicol, “Interaction of gold(I) with thiosulfate-sulfite mixed ligand systems.”, *Inorg. Chim. Acta*, vol 358, pp 2183- 2190, 2005

W N Perera, G Senanayake and M J Nicol, “A study of the gold colloid dissolution kinetics in oxygenated ammoniacal thiosulfate solutions”, *Hydrometallurgy*, vol 74, pp 243-257, 2005

H Zhang, M J Nicol and W P Staunton, “An electrochemical study of an alternative process for the leaching of gold in thiosulfate solutions.” *Treatment of Gold Ores, Proceedings of the International Symposium on the Treatment of Gold Ore*, Calgary, AB, Canada, Aug. 2005 (2005), pp243-257.

C Van Wensveen, and M J Nicol, “The kinetics of the copper-catalysed oxidation of thiosulfate in ammoniacal solutions.”, *Treatment of Gold Ores, Proceedings of the International Symposium on the Treatment of Gold Ore*, Calgary, AB, Canada, Aug. 2005 (2005), pp193-207.

A N Nikoloski and M J Nicol, “The electrochemistry of the leaching reactions in the Caron process. I. anodic processes.”, *Electrochemistry in Mineral and Metal Processing VII*, The Electrochemical Society, Pennington, NJ, pp 197-207, 2006.

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M S Lee and M J Nicol, “Removal of iron from cobalt sulfate solutions by ion exchange with Diphonix resin and enhancement of iron elution with titanium(III).” *Hydrometallurgy*, vol 86, pp 6-12, 2007.

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A Nikoloski and M J Nicol, “ The effect of cobalt ions on the performance of lead anodes used for the electrowinning of copper – A literature review”, *Mineral Processing & Extractive Metall. Rev*, vol 29, pp 143-172, 2008

M S Lee, M J Nicol and P Basson, “Cathodic Processes in the Leaching and Electrochemistry of Covellite in Mixed Sulfate - Chloride Media”, *Journal of Applied Electrochemistry* vol 38, pp 363-369, 2008.

T Nguyen, N Guresin, M Nicol and A Atrens, "Influence of cobalt ions on the anodic oxidation of a lead alloy under conditions typical of copper electrowinning", *Journal of Applied Electrochemistry* vol 38, pp 215-224, 2008.

M S Lee and M J Nicol, "Ionic Equilibria in Mixed Solutions of Cuprous and Cupric Chloride", *J. Kor. Inst. Met. & Mater.*, vol 46, pp 20-25, 2008.

H Miki and M J Nicol, "Synergism in the oxidation of covellite and pyrite in chloride solutions containing iron(III) and copper(II)", *Hydrometallurgy* 2008, Phoenix, pp 646-652, August 2008.

H Miki and M J Nicol, "The Kinetics of the Copper-catalysed Oxidation of Iron(II) in Chloride Solutions", *Hydrometallurgy* 2008, Phoenix, pp 971-979, August 2008.

McGinnity, J and Nicol, M, "The Effects of Periodic Open-Circuit on the Corrosion of Lead Alloy Anodes in Sulfuric Acid Electrolytes containing Manganese", *Hydrometallurgy* 2008, Phoenix, pp 592-599, August 2008.

A Senaputra, G Senanayake, M J Nicol, and A Nikoloski, "Leaching nickel and nickel sulfides in ammonia/ammonium carbonate solutions", *Hydrometallurgy* 2008, Phoenix, pp 551-560, August 2008.

E Roux and M J Nicol, "The Effects of Tangential or Perpendicular Inlet Flow on Mass Transfer in an Annular Electrochemical Cell", *Hydrometallurgy* 2008, Phoenix, pp 625-631, August 2008.

X M Zhang, G Senanayake and M J Nicol, "Beneficial effect of silver in thiosulfate leaching of gold", *Hydrometallurgy* 2008, Phoenix, pp 801-810, August 2008.

Z Zainol and M J Nicol, "Comparative study of chelating ion exchange resins for the recovery of nickel and cobalt from laterite leach tailings", *Hydrometallurgy*, vol 96, pp 283-287, 2009

S Zhang and M J Nicol, "An Electrochemical Study of the Reduction and Dissolution of Ilmenite in Sulfuric Acid Solutions", *Hydrometallurgy*, vol 97, pp 146-157, 2009

Z Zainol and M J Nicol, "Ion-exchange equilibria of Ni^{2+} , Co^{2+} , Mn^{2+} and Mg^{2+} with iminodiacetic acid chelating resin, Amberlite IRC 748", *Hydrometallurgy*, vol 99, pp 175-180, 2009

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S Zhang and M J Nicol, "Kinetics of the Dissolution of Ilmenite in Sulphuric Acid Under Reducing Conditions", *Hydrometallurgy*, *in press*

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L Velásquez-Yévenes, M J Nicol, and H Miki , "The dissolution of chalcopyrite in chloride solutions. 1. The effect of solution potential", *Hydrometallurgy*.

L Velásquez Yévenes, H Miki and M J Nicol , "The dissolution of chalcopyrite in chloride solutions. 2. Effect of various parameters on the rate." *Hydrometallurgy*

L Velásquez Yévenes, H Miki and M J Nicol , "The dissolution of chalcopyrite in chloride solutions. 3. Mechanisms." *Hydrometallurgy*

UNREFERREED CONFERENCE PRESENTATIONS

Nicol, M.J. "Trends in the Kinetics and Mechanisms of the Dissolution of Sulfide Minerals", 1999 Parker Centre Conference on Leaching Processes, Perth, October 1999.

Nicol, M.J., Lallenec, S. and Zainol, Z, "Development of a Resin-in-Pulp Process for the Recovery of Nickel and Cobalt from Laterite Leach Slurries", 2001 Parker Centre Conference on Separation Processes, Perth, October 2001.

Nicol, M.J., "The mechanisms of the leaching of sulphide minerals – a re-appraisal", VII Meeting of the Southern Hemisphere on Mineral Technology, Auro Preto, Minas Gerais, Brazil, November, 2007.

Professional Development Courses

Responsibility for the delivery and assessment (in some cases) of the following recent courses

MSc in Hydrometallurgy, University of Cape Town, July 2003 and Jan 2004 : July 2005 and Jan 2006: Jan 2008 and July 2008, Jan 2009 and July 2009.

Electrowinning of Zinc, Teck Cominco, Sep 2003

Electrowinning of Copper, Phelps Dodge, Arizona, Oct 2003 and Feb 2005

Fundamentals of Hydrometallurgy, Amplats, Rustenburg, Jan 2005
Electrowinning of Metals, Belo Horizonte, Brazil, April 2005
Electrowinning of Copper, BHPBilliton, Santiago, April 2005
Fundamentals of Hydrometallurgy, BHPBilliton, Newcastle, August 2005
Electrowinning of Metals, Brisbane, November 2005
Electrowinning of Metals, Anglo-American Corp, Johannesburg, February, 2006
Hydrometallurgy, Anglo-American Corp, Johannesburg, July 2006
Leaching and Separation Processes, Brisbane, November 2006
Fundamentals of Hydrometallurgy, Rio Tinto, Melbourne, July 2007
Electrowinning of Metals, Teck Cominco - CESL, Vancouver, Sep 2007
Electrowinning of Copper, Freeport-MacMoran, Arizona, Sep 2007
Fundamentals of Hydrometallurgy, BHPBilliton, Newcastle, Nov 2007
MSc in Hydrometallurgy, University of Cape Town, Jan 2008
Fundamentals of Hydrometallurgy, Olympic Dam, S. Australia, Feb 2008
Electrowinning of Copper, Antofagasta, Chile, April, 2008
Hydrometallurgy, Anglo-American Corp, Johannesburg, December, 2008
MSc in Hydrometallurgy, University of Cape Town, Jan and July 2009
Ion Exchange Processes, AngloResearch, July 2009-10-24



Professor Michael J Nicol

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : Christopher Brett Ward
Serial Number : 10/501,277
Filing Date : September 3, 2004
Title : Hydrometallurgical Processing of
Manganese Containing Materials
Examiner : Arun S. Phasge
Group Art Unit : 1795

DECLARATION OF MICHAEL J. NICOL UNDER 37 C.F.R. §1.132

This is Annexure MJN-2 referred to in my Declaration made this 22 day
of FEBRUARY 2010.



Professor Michael J Nicol

"Hydrometallurgical Processing of Manganese Containing Materials"

Field of the Invention

The present invention relates to the hydrometallurgical processing of manganese containing materials. More particularly, the process of the present invention is intended to allow efficient hydrometallurgical processing of low-grade manganese dioxide feedstock to produce manganese products, including electrolytic manganese dioxide.

Background Art

It is generally known that manganese may be leached from manganese dioxide containing ores using sulphur dioxide. However, the sulphur dioxide leaching of manganese dioxide containing materials is also known to produce by-product dithionate ion levels of $>5\text{g/l}$. These levels may be far higher depending upon the amount of manganese being leached. For example, levels of about 20g/l are not uncommon. Interestingly, it is reported in Cotton and Wilkinson, Advanced Inorganic Chemistry, 3rd Edition at page 452, that "the method for production of dithionate or dithionic acid is the reaction between sulphur dioxide or sulphite with manganese dioxide in the presence of acid".

Low grade manganese dioxide feedstock ($<40\%$ Mn) are presently uneconomic to process using conventional roast-reduction and sulphuric acid leaching to produce manganese sulphate. High grade ores ($>40\%$ Mn) are needed to justify the economics of the roast reduction process. Presently, all leaching of manganese dioxide containing materials using sulphur dioxide leads to the formation of $>5\text{g/l}$ levels of dithionate ions in solution. With dithionate ion levels of this magnitude it is generally necessary to incorporate into any flow sheet a high capital cost stage, being "oxidation" or "aging". The long residence times required to "oxidise" the dithionate ion from the $>5\text{g/l}$ levels down to lower than 1g/l are highly capital intensive.

Failure to control dithionate levels in the production of a manganese sulphate crystal product has previously led to the manganese dithionate contaminant in that product slowly reacting to release sulphur dioxide gas.

5 It would prove advantageous to provide a process whereby low-grade manganese dioxide containing materials or feedstock could provide manganese sulphate leach solutions with a level of dithionate ion less than about 5g/l, and preferably less than 1g/l.

10 The ability to recover manganese dioxide from low-grade feedstocks will avoid or at least reduce the need for further manganese ore mining and land disturbance, bringing various environmental benefits. For example, the utilisation of manganese tailings allows for conservation of existing resources.

Further, the use of a hydrometallurgical route for the reduction of Mn(IV) negates the need for the use of gas fired kilns or fluid bed reactors, feed stocks no longer need to be heated to about 1000°C and then cooled prior to leaching, and there
15 is lesser need for carbon input, which in turn results in lower greenhouse gas emissions.

Still further, the use of the relatively easily controlled hydrometallurgical route allows monitoring of the solution potential of the leach solution or slurry thereby indicating complete dissolution of Mn(IV). The use of the sulphur dioxide leach
20 provides complete conversion of Mn(IV) to Mn(II), thereby avoiding the production of leachable manganese species in solid residues.

In particular, if it is desired to produce electrolytic manganese dioxide ("EMD"), solutions containing elevated dithionate ion levels result in chemical reactions occurring that effect the quality and purity of the EMD produced in the
25 electrowinning cells. Also, hydrogen sulphide is evolved, bringing with it certain occupational health and environmental issues.

The preceding discussion of the background art is intended to facilitate an understanding of the present invention only. It should be appreciated that the

discussion is not an acknowledgement or admission that any of the material referred to was part of the common general knowledge in Australia as at the priority date of the application.

Throughout the specification, unless the context requires otherwise, the word
5 "comprise" or variations such as "comprises" or "comprising", will be understood to imply the inclusion of a stated integer or group of integers but not the exclusion of any other integer or group of integers.

Disclosure of the Invention

C In accordance with the present invention there is provided a process for the
10 hydrometallurgical processing of manganese containing materials, the process characterised by the combination of a manganese dioxide containing feedstock and an acidic solution to form a leach solution, and passing a volume of sulphur dioxide gas through that leach solution, whereby the levels of dithionate ion generated in the leach solution are less than about 5g/l.

15 Preferably, the levels of dithionate ion generated in the leach solution are less than about 1g/l.

Still preferably, the pH of the leach solution is maintained at less than about 1.5.

C In one form of the invention the leach solution comprises a slurry of manganese
20 dioxide containing material at a slurry density of less than about 10%w/v, less than about 120g/l manganese sulphate, a temperature of greater than about 95°C, and at a pH of less than about 1.5.

In another form of the invention the manganese dioxide containing feedstock contains less than about 40% manganese.

Preferably, the leach solution has an initial soluble iron concentration of greater
25 than 4g/l. The iron is preferably in the form of ferric sulphate ($\text{Fe}_2(\text{SO}_4)_3$). The

ferrous concentration is preferably maintained at a level below about 0.5g/l by providing an excess or residual amount of manganese dioxide in the slurry.

- 5 The ratio of ferric to ferrous may be monitored throughout the leach to ensure an oxidation reduction potential (ORP) of 550mV, or above (vs Ag/AgCl reference electrode).

The sulphur dioxide gas is preferably passed through the leach solution over a period of at least 10 hours whereby up to about 95% of manganese dioxide is dissolved. Preferably, the leach is conducted over a period of between about 10 to 15 hours.

- 10 Preferably, once a stoichiometric amount of sulphur dioxide has been added to the leach solution to achieve a 95% dissolution of the manganese dioxide present, the reaction is halted.

- 15 In accordance with present invention there is further provided a process for the production of electrolytic manganese dioxide, the process characterised by a leach of a manganese dioxide containing feedstock in acidic solution, through which a volume of sulphur dioxide gas is passed, and in which dithionate ion levels are maintained at less than about 5g/l, the resulting leach solution being processed to provide an appropriate electrolyte that is passed to an electrowinning stage during which electrolytic manganese dioxide is deposited.

- 20 Preferably, the levels of dithionate ion generated in the leach solution are less than about 1g/l.

Still preferably, the pH of the leach solution is maintained at less than about 1.5.

- 25 In one form of the invention the leach solution comprises a slurry of manganese dioxide containing material at a slurry density of less than about 10%w/v, less than about 120g/l manganese sulphate, a temperature of greater than about 95°C, and at a pH of less than about 1.5.

In another form of the invention the manganese dioxide containing feedstock contains less than 40% manganese.

Preferably, the leach solution has an initial soluble iron concentration of greater than 4g/l. The iron is preferably in the form of ferric sulphate ($\text{Fe}_2(\text{SO}_4)_3$). The ferrous concentration is preferably maintained at a level below about 0.5g/l by providing an excess or residual amount of manganese dioxide in the slurry.

The ratio of ferric to ferrous may be monitored throughout the leach to ensure an oxidation reduction potential (ORP) of 550mV, or above (vs Ag/AgCl reference electrode).

10 The sulphur dioxide gas is preferably passed through the leach solution over a period of at least 10 hours whereby up to about 95% of manganese dioxide is dissolved. Preferably, the leach is conducted over a period of between about 10 to 15 hours.

15 Preferably, once a stoichiometric amount of sulphur dioxide has been added to the leach solution to achieve a 95% dissolution of the manganese dioxide present, the reaction is halted.

20 Preferably, the acidic solution used in the leach is at least in part comprised of return or spent sulphuric acid solution from the electrowinning stage. It may be necessary to add additional acid to the leach to ensure the pH remains less than about 1.5.

Brief Description of the Drawings

The present invention will now be described, by way of example only, with reference to one embodiment thereof and the accompanying drawings, in which:-

25 Figure 1 is a schematic flow chart of a process for the production of an electrolytic manganese dioxide product from a low grade manganese feedstock in accordance with the present invention;

Figure 2 is a graphical representation of the percentage manganese "leached" relative to SO_2 addition and Eh during the leach conducted within the process of Figure 1; and

5 Figure 3 is a graphical representation of the percentage manganese "leached" over time and relative to SO_2 additional during the leach of Figure 2.

Best Mode(s) for Carrying Out the Invention

C In Figure 1 there is shown a process 10 for the production of electrolytic manganese dioxide (EMD) product in accordance with the present invention. A
10 manganese dioxide ore feedstock 12, containing about 34% manganese, is initially milled in a wet ball mill 14 prior to the generated slurry being passed to a leach 16 conducted in at least one agitated reactor.

In addition to the milled feedstock slurry the leach 16 is fed with return sulphuric acid electrolyte 17 from an electrowinning stage 18 to form a leach solution. The
15 leach solution has a volume of sulphur dioxide gas 20 passed therethrough, the sulphur dioxide gas 20 being formed through a sulphur burning step 22, in which sulphur is burnt in oxygen to generate sulphur dioxide.

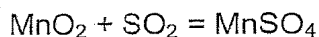
C The slurry density of the feedstock fed from the mill 14 is less than 10% w/v. The slurry is both heated and agitated, as the leach solution, once within the or each
20 reactor 16. The concentration of manganese sulphate (MnSO_4) in the leach solution is $<120\text{g/l}$ whilst the temperature of the leach solution is $>95^\circ\text{C}$. The leach solution is maintained at a pH of less than about 1.5.

The leach solution within the leach 16 has an initial soluble iron concentration of $>4\text{g/l}$. The iron is in the form of ferric sulphate ($\text{Fe}_2(\text{SO}_4)_3$). The ferrous
25 concentration is maintained at less than 0.5g/l by ensuring there is always an excess or residual amount of manganese dioxide in the leach.

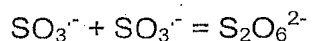
The leach is conducted over a period of 10 to 15 hours by passing a volume of sulphur dioxide gas through the leach solution. The rate at which the sulphur dioxide is passed through the leach solution is governed by the requirement that up to 95% of the manganese dioxide in the slurry fed to the leach 16 is dissolved/solubilized over the period of 10 to 15 hours. It is envisaged that longer time periods may be utilised.

The ratio of ferric to ferrous in the leach 16 is monitored by an Oxidation Reduction Potential (ORP) probe. A minimum value of 550mV is required (vs Ag/AgCl reference electrode). If the ORP value falls below 550mV more feedstock slurry is added to the leach 16.

It is believed that the predominant leaching reaction proceeds as follows:



However, it is understood that whilst the process of the present invention ensures that the production of manganese sulphate is the predominant reaction, a trace of dithionate is still generated. The production of dithionate is believed to proceed as a free radical combination reaction as follows:



High pH values favour the production of dithionate. As such, the pH of the leach 16 is maintained at less than about 1.5, as noted hereinabove.

Once a stoichiometric quantity of sulphur dioxide 20 has been fed through the leach solution, whereby a maximum of 95% of the manganese dioxide present is dissolved/solubilized, the reaction is halted by withdrawing the sulphur dioxide 20 feed to the leach 16. The stoichiometric addition of SO_2 is represented graphically in Figure 2 relative to the % manganese leached and the Eh, showing that the Eh or solution potential provides an accurate indication of completion of the manganese (IV) dissolution reaction, whilst Figure 3 demonstrates the effect of sulphur dioxide addition on the % manganese leached over time.

It is envisaged that the sulphur dioxide 20 might also be provided as a waste gas from a smelting or an industrial process. Further, the sulphur dioxide might be added to the leach solution as a sulphite solution (SO_3^{2-}).

5 The remainder of the process 10 for the production of EMD involves the passing of pregnant leach solution to a jarositing step 22 to reduce potassium and sodium levels as desired. A goethiting step 24 is then used to reduce iron levels to sub-ppm levels through the addition of ground limestone. A solid/liquid separation step is then used, involving both thickening 26 and filtration 28.

10 Solids from the filtration 28 are washed to reclaim any residual manganese sulphate solution, this being fed back to the leach 16 to make up any volume lost from the return electrolyte 17 to a manganese sulphate bleed stream 30. This bleed stream 30 allows manganese sulphate to be concurrently produced for use in fertilisers, for example.

15 Overflow from the thickening 26 is passed to a sulphiding step 32 for the removal of heavy metals, including nickel, cobalt and molybdenum. Following the sulphiding step 32 the heavy metal sulphides are removed by pressure filtration 34 and the electrolyte from the electrowinning step 18 held in storage tanks 36.

20 The electrowinning step 18 then proceeds utilising submerged titanium anodes, tubular copper cathodes and a totally wax free environment. Fully laden anodes are harvested on a two-weekly cycle with resulting EMD chip being passed to produce processing and packaging operations.

25 It is envisaged that the process of the present invention, specifically as it resides in the leaching of manganese containing feedstocks to produce leach solutions with dithionate ion levels of less than 5g/l, and preferably less than 1g/l, may be applied to the processing of all types of manganese dioxide containing ores (including both high and low grade), mine tailings, fines, fumes and tailings of manganese ferro-alloy production facilities, ocean floor manganese nodules, ferromanganese nodules, wastes from zinc refinery cells and manganese dioxide contained in used or partially used alkaline or carbon zinc batteries. The leach

solutions generated by reprocessing such materials in accordance with the present invention can then be purified and used in the production of EMD, EMM and other manganese chemical products.

Modifications and variations such as would be apparent to the skilled addressee
5 are considered to fall within the scope of the present invention.

AMENDMENTS TO THE CLAIMS:

29. (Currently Amended) A process for the hydrometallurgical processing of manganese containing materials, the process characterised by the combination of a manganese dioxide containing feedstock and an acidic solution to form an acidic solution to be leached, and passing a volume of sulphur dioxide gas through that leach solution as the leaching agent, whereby no sintering or roasting pre-treatment step of the feedstock is undertaken and the levels of dithionate ion generated in the leach solution are less than about 5g/l.

30. (Previously Amended) A process according to claim 29, wherein the levels of dithionate ion generated in the leach solution are less than about 1g/l.

31. (Previously Amended) A process according to claim 29, wherein the pH of the leach solution is maintained at less than about 1.5.

32. (Previously Amended) A process according to claim 29, wherein the leach solution comprises a slurry of manganese dioxide containing material at a slurry density of less than about 10%w/v, less than about 120g/l manganese sulphate, a temperature of greater than about 95°C, and at a pH of less than about 1.5.

33. (Previously Amended) A process according to claim 29, wherein the manganese dioxide containing feedstock contains less than about 40% manganese.
34. (Previously Amended) A process according to claim 29, wherein the leach solution has an initial soluble iron concentration of greater than 4g/l.
35. (Previously Amended) A process according to claim 34, wherein the iron is in the form of ferric sulphate ($\text{Fe}_2(\text{SO}_4)_3$).
36. (Previously Amended) A process according to claim 29, wherein the ferrous concentration is maintained at a level below about 0.5g/l by providing an excess or residual amount of manganese dioxide in the slurry.
37. (Previously Amended) A process according to claim 29, wherein the ratio of ferric to ferrous is monitored at least at intervals throughout the leach to ensure an oxidation reduction potential (ORP) of 550mV, or above (vs Ag/AgCl reference electrode).
38. (Previously Amended) A process according to claim 29, wherein the sulphur dioxide gas is preferably passed through the leach solution over a period of at least 10 hours whereby up to about 95% of manganese dioxide is dissolved.

39. (Previously Amended) A process according to claim 29, wherein the leach is conducted over a period of between about 10 to 15 hours.

40. (Previously Amended) A process according to claim 29, wherein once a stoichiometric amount of sulphur dioxide has been added to the leach solution to achieve a 95% dissolution of the manganese dioxide present, the reaction is halted.

41. (Currently Amended) A process for the production of electrolytic manganese dioxide, the process characterised by a leach of a manganese dioxide containing feedstock, without a sintering or roasting pre-treatment step of that feedstock, in which a volume of sulphur dioxide gas as the leaching agent is passed through an acidic solution containing manganese dioxide, the dithionate ion levels in said solution being maintained at less than about 5g/l, the resulting leach solution being processed to provide an appropriate electrolyte that is passed to an electrowinning stage during which electrolytic manganese dioxide is deposited.

42. (Previously Amended) A process according to claim 41, wherein the levels of dithionate ion generated in the leach solution are less than about 1g/l.

43. (Previously Amended) A process according to claim 41, wherein the pH of the leach solution is maintained at less than about 1.5.

44. (Previously Amended) A process according to claim 41, wherein the leach solution comprises a slurry of manganese dioxide containing material at a slurry density of less than about 10%w/v, less than about 120g/l manganese sulphate, a temperature of greater than about 95°C, and at a pH of less than about 1.5.

45. (Previously Amended) A process according to claim 41, wherein the manganese dioxide containing feedstock contains less than 40% manganese.

46. (Previously Amended) A process according to claim 41, wherein the leach solution has an initial soluble iron concentration of greater than 4g/l.

47. (Previously Amended) A process according to claim 46, wherein the iron is in the form of ferric sulphate ($\text{Fe}_2(\text{SO}_4)_3$).

48. (Previously Amended) A process according to claim 41, wherein the ferrous concentration is maintained at a level below about 0.5g/l by providing an excess or residual amount of manganese dioxide in the slurry.

49. (Previously Amended) A process according to claim 41, wherein the ratio of ferric to ferrous is monitored throughout the leach to ensure an oxidation reduction potential (ORP) of 550mV, or above (vs Ag/AgCl reference electrode).

50. (Previously Amended) A process according to claim 41, wherein the sulphur dioxide gas is passed through the leach solution over a period of at least 10 hours whereby up to about 95% of manganese dioxide is dissolved.

51. (Previously Amended) A process according to claim 41, wherein the leach is conducted over a period of between about 10 to 15 hours.

52. (Previously Amended) A process according to claim 41, wherein once a stoichiometric amount of sulphur dioxide has been added to the leach solution to achieve a 95% dissolution of the manganese dioxide present, the reaction is halted.

53. (Previously Amended) A process according to claim 41, wherein the acidic solution used in the leach is at least in part comprised of return or spent sulphuric acid solution from the electrowinning stage.

54. (Previously Amended) A process according to claim 41, wherein additional acid is added to the leach to ensure the pH remains less than about 1.5.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : Christopher Brett Ward
Serial Number : 10/501,277
Filing Date : September 3, 2004
Title : Hydrometallurgical Processing of
Manganese Containing Materials
Examiner : Arun S. Phasge
Group Art Unit : 1795

DECLARATION OF MICHAEL J. NICOL UNDER 37 C.F.R. §1.132

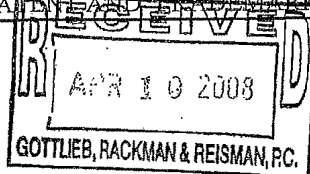
This is Annexure MJN-3 referred to in my Declaration made this 22 day
of FEBRUARY 2010.



Professor Michael J Nicol



UNITED STATES PATENT AND TRADEMARK OFFICE



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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/501,277	09/03/2004	Christopher Brett Ward	4046-022	5676

22440 7590 04/04/2008
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EXAMINER PHASGE, ARUN S	
ART UNIT 1795	PAPER NUMBER
MAIL DATE 04/04/2008	DELIVERY MODE PAPER

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

Office Action Summary	Application No.	Applicant(s)	
	10/501,277	WARD, CHRISTOPHER BRETT	
	Examiner	Art Unit	
	Arun S. Phasge	1795	

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) OR THIRTY (30) DAYS, WHICHEVER IS LONGER, FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
 - If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
 - Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133).
- Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

Status

- 1) ☐ Responsive to communication(s) filed on ____.
- 2a) ☐ This action is **FINAL**. 2b) ☒ This action is non-final.
- 3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

Disposition of Claims

- 4) ☒ Claim(s) 29-52 is/are pending in the application.
- 4a) Of the above claim(s) ____ is/are withdrawn from consideration.
- 5) ☐ Claim(s) ____ is/are allowed.
- 6) ☒ Claim(s) 29-52 is/are rejected.
- 7) ☐ Claim(s) ____ is/are objected to.
- 8) ☐ Claim(s) ____ are subject to restriction and/or election requirement.

Application Papers

- 9) ☐ The specification is objected to by the Examiner.
- 10) ☐ The drawing(s) filed on ____ is/are: a) ☐ accepted or b) ☐ objected to by the Examiner.
Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).
Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).
- 11) ☐ The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

Priority under 35 U.S.C. § 119

- 12) ☒ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
- a) ☒ All b) ☐ Some * c) ☐ None of:
1. ☒ Certified copies of the priority documents have been received.
 2. ☐ Certified copies of the priority documents have been received in Application No. ____.
 3. ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

* See the attached detailed Office action for a list of the certified copies not received.

Attachment(s)

- | | |
|--|---|
| 1) <input checked="" type="checkbox"/> Notice of References Cited (PTO-892) | 4) <input type="checkbox"/> Interview Summary (PTO-413) |
| 2) <input type="checkbox"/> Notice of Draftsperson's Patent Drawing Review (PTO-948) | Paper No(s)/Mail Date. ____. |
| 3) <input checked="" type="checkbox"/> Information Disclosure Statement(s) (PTO/SB/08) | 5) <input type="checkbox"/> Notice of Informal Patent Application |
| Paper No(s)/Mail Date <u>7/9/04</u> . | 6) <input type="checkbox"/> Other: ____. |

Application/Control Number: 10/501,277
Art Unit: 1795

Page 2

DETAILED ACTION

Claim Objections

The numbering of claims is not in accordance with 37 CFR 1.126 which requires the original numbering of the claims to be preserved throughout the prosecution. When claims are canceled, the remaining claims must not be renumbered. When new claims are presented, they must be numbered consecutively beginning with the number next following the highest numbered claims previously presented (whether entered or not).

Misnumbered claims 1-24 been renumbered 29-52.

Claim Rejections - 35 USC § 103

The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

The factual inquiries set forth in *Graham v. John Deere Co.*, 383 U.S. 1, 148 USPQ 459 (1966), that are applied for establishing a background for determining obviousness under 35 U.S.C. 103(a) are summarized as follows:

1. Determining the scope and contents of the prior art.
2. Ascertaining the differences between the prior art and the claims at issue.
3. Resolving the level of ordinary skill in the pertinent art.
4. Considering objective evidence present in the application indicating obviousness or nonobviousness.

Claims 29-52 are rejected under 35 U.S.C. 103(a) as being unpatentable over Kasaian, U.S. Patent 5,932,086 in view of Leonard, U.S. Patent 3,301,777.

The Kasaian patent discloses the claimed method for the hydrometallurgical processing of manganese containing materials, the process characterized by the combination of a manganese dioxide containing feedstock and an acidic solution to form a leach solution, and passing a volume of sulfur dioxide gas through the leach solution and the claimed pH (see examples 1-2 in columns 4-5). The reference further discloses the same relative concentrations, temperature and time as claimed (see examples 1-4).

Normally, change in temperature, concentration, or both, is not patentable modification; however, such changes may impart patentability to process if ranges claimed produce new and unexpected result which is different in kind and not merely in degree from results of prior art; such ranges are termed "critical" ranges, and applicant has burden of proving such criticality; even though applicant's modification results in great improvement and utility over prior art, it may still not

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Art Unit: 1795

Page 4

be patentable if modification was within capabilities of one skilled in art; more particularly, where general conditions of claim are disclosed in prior art, it is not inventive to discover optimum or workable ranges by routine experimentation. In re Aller et al, 105 U.S.P.Q. 233 CCPA (1955).

The patent does not disclose that the concentrations of dithionate ions are kept below 5 g/l or less than about 1 g/l, although it states that sufficient sulfur dioxide is added to allow the process to occur (see col. 3, lines 30-38).

The Leonard patent is cited to show the use of the range recited in the claims (see col. 4, lines 39-50).

Consequently, the invention as a whole would have been obvious to one having ordinary skill in the art at the time the invention was made to modify the disclosure of the Kasaaian patent with the teachings of the Leonard patent, because the Leonard patent teaches the use of the claimed range of dithionate ions.

Conclusion

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Arun S. Phasge whose telephone number is

Application/Control Number: 10/501,277
Art Unit: 1795

Page 5

(571) 272-1345. The examiner can normally be reached on MONDAY-THURSDAY, 7:30-6:00.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Nam X. Nguyen can be reached on (571) 272-1342. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

Application/Control Number: 10/501,277
Art Unit: 1795

Page 6

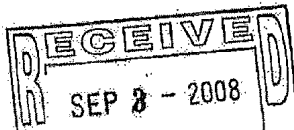
/Arun S. Phasge/
Primary Examiner, Art Unit 1795

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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/501,277	09/03/2004	Christopher Brett Ward	4046-022	5676

22440 7590 10/30/2008
GOTTLIEB RACKMAN & REISMAN PC
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8TH FLOOR
NEW YORK, NY 10016-0601



EXAMINER	
PHASGE, ARUN S	
ART UNIT	PAPER NUMBER
1795	
MAIL DATE	DELIVERY MODE
10/30/2008	PAPER

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

Office Action Summary	Application No.	Applicant(s)	
	10/501,277	WARD, CHRISTOPHER BRETT	
	Examiner	Art Unit	
	Arun S. Phasga	1795	

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) OR THIRTY (30) DAYS, WHICHEVER IS LONGER, FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

Status

- 1) ☒ Responsive to communication(s) filed on 30 July 2008.
- 2a) ☐ This action is **FINAL**. 2b) ☒ This action is non-final.
- 3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

Disposition of Claims

- 4) ☒ Claim(s) 29-54 is/are pending in the application.
- 4a) Of the above claim(s) _____ is/are withdrawn from consideration.
- 5) ☐ Claim(s) _____ is/are allowed.
- 6) ☒ Claim(s) 29-54 is/are rejected.
- 7) ☐ Claim(s) _____ is/are objected to.
- 8) ☐ Claim(s) _____ are subject to restriction and/or election requirement.

Application Papers

- 9) ☐ The specification is objected to by the Examiner.
- 10) ☐ The drawing(s) filed on _____ is/are: a) ☐ accepted or b) ☐ objected to by the Examiner.
 Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).
 Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).
- 11) ☐ The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

Priority under 35 U.S.C. § 119

- 12) ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
 a) ☐ All b) ☐ Some * c) ☐ None of:
1. ☐ Certified copies of the priority documents have been received.
2. ☐ Certified copies of the priority documents have been received in Application No. _____.
3. ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).
- * See the attached detailed Office action for a list of the certified copies not received.

Attachment(s)

- | | |
|--|---|
| 1) <input type="checkbox"/> Notice of References Cited (PTO-892) | 4) <input type="checkbox"/> Interview Summary (PTO-413) |
| 2) <input type="checkbox"/> Notice of Draftsperson's Patent Drawing Review (PTO-948) | Paper No(s)/Mail Date: _____ |
| 3) <input type="checkbox"/> Information Disclosure Statement(s) (PTO/SB/08) | 5) <input type="checkbox"/> Notice of Informal Patent Application |
| Paper No(s)/Mail Date: _____ | 6) <input type="checkbox"/> Other: _____ |

DETAILED ACTION

The text of those sections of Title 35, U.S. Code not included in this action can be found in a prior Office action.

Claim Rejections - 35 USC § 103

Claims 20-54 are rejected under 35 U.S.C. 103(a) as being unpatentable over Kasaaian in view of Leonard of record for reasons of record.

The use of the spent sulfuric acid from the electrolysis stage as the sulfuric acid used in the leach as claimed in claim 53 would have been an obvious embodiment to the ordinary artisan motivated by the economic considerations to re-use sulfuric acid.

The Leonard patent teaches the maintenance of the concentrations within the range claimed in claim 54. Accordingly, the claims as a whole would have been obvious to one having ordinary skill in the art at the time the invention was made, because the Leonard patent teaches the range of concentration used in the art to electrowin manganese.

Response to Arguments

Applicant's arguments filed 7/30/08 have been fully considered but they are not persuasive.

Applicants argue that the prior art differs from the instant claimed method by stating, "By contrast, in the prior art (Kasaaian), the leach is conducted with sulphuric acid, not with sulphur dioxide. Kasaaian actually describes a process of sintering of manganese dioxide ore, which is fundamentally different from the leach of the present invention. This sintering of manganese dioxide ore first converts the lower oxides of

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manganese contained in the ore (MnO_2 and Mn_2O_3) to manganomanganic oxide (Mn_3O_4), by using sulphur dioxide as a reducing agent; this takes place before the Mn_3O_4 is subsequently leached with sulphuric acid. As such, the prior art teaches leaching with sulphuric acid only *not* with sulphur dioxide, which is used there only as a reducing agent."

Example 1 of the Kasaian patent teaches a leach process that comprises the use of sulfuric acid with sulphur dioxide bubbled through the tank (see columns 4 and 5 showing example 1).

Therefore, the examiner's reading that the "Kasaian patent discloses the claimed method for the hydrometallurgical processing of manganese containing materials is correct.

Applicants next attack the combination by stating, "the Examiner's citation of the Leonard patent, allegedly to supply that "missing" element, also evinces an unfortunate misunderstanding, Leonard simply suggests, at column 4, lines 39-42, that an amount of dithionate may be "included in the electrolyte (sic)"; in other words, Leonard suggests that dithionate be introduced into the reaction mixture as a starting material, *before* the sulphur dioxide gas is passed through the mixture and the leach reaction takes place. On the other hand, in applicant's claimed method, the dithionate ions are products (or, more aptly, by-products) of the leaching reaction, as can be understood from the specification, when taken together with the use of the phrase "generated in the leach solution" in applicant's independent claim 29.

Therefore, it is the amount of dithionate formed as a reaction product, *not* the amount used as a starting material, which applicant has realized must be controlled, in the context of a leach reaction for manganese containing materials that utilizes sulphur dioxide, not sulphuric acid, as the leaching agent. As such, the combination of Kasaian and Leonard in the manner postulated by the Examiner (that is, by "modifying" the Kasaian disclosure with the teachings of Leonard) is inappropriate and would certainly not be a combination that a skilled worker in this field would arrive at or consider in this context, and in any event, would not yield applicant's invention as presently claimed."

The Leonard patent states in col. 4, beginning in line 33, "When the present invention is utilized in the sulfate process for the production of electrolytic manganese according to one preferred procedure, the **operating variables** can be conveniently adjusted within the following limits." The patent does not state as unfortunately inferred by applicants, that the amount of the dithionate is used as a starting material, since these values are operating variables. Indeed, the whole paragraph is the control of different limits during the process and not merely at the start of the process, since the reference does not even use the words "as a starting material."

Consequently, the invention as a whole would have been obvious to one having ordinary skill in the art at the time the invention was made to use the clear teachings of Leonard to control the amount of the dithionite in a sulfate process for the electrolytic formation of electrolytic manganese.

Conclusion

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Arun S. Phasge whose telephone number is (571) 272-1345. The examiner can normally be reached on MONDAY-THURSDAY, 7:30-6:00.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Nam X. Nguyen can be reached on (571) 272-1342. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Application/Control Number: 10/501,277
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Page 5

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/Arun S. Phasge/
Primary Examiner, Art Unit 1795

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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/501,277	09/03/2004	Christopher Brett Ward	4046-022	5676

22440 7590 04/14/2009
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8TH FLOOR
NEW YORK, NY 10016-0601

EXAMINER

PHASGE, ARUN S

ART UNIT	PAPER NUMBER
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1795

MAIL DATE	DELIVERY MODE
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04/14/2009

PAPER

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

Office Action Summary

Application No.

10/501,277

Applicant(s)

WARD, CHRISTOPHER BRETT

Examiner

Arun S. Phasge

Art Unit

1795

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) OR THIRTY (30) DAYS, WHICHEVER IS LONGER, FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

Status

- 1) ☒ Responsive to communication(s) filed on 23 January 2009.
- 2a) ☒ This action is FINAL. 2b) ☐ This action is non-final.
- 3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

Disposition of Claims

- 4) ☒ Claim(s) 29-54 is/are pending in the application.
- 4a) Of the above claim(s) _____ is/are withdrawn from consideration.
- 5) ☐ Claim(s) _____ is/are allowed.
- 6) ☒ Claim(s) 29-54 is/are rejected.
- 7) ☐ Claim(s) _____ is/are objected to.
- 8) ☐ Claim(s) _____ are subject to restriction and/or election requirement.

Application Papers

- 9) ☐ The specification is objected to by the Examiner.
- 10) ☐ The drawing(s) filed on _____ is/are: a) ☐ accepted or b) ☐ objected to by the Examiner.
Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).
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- 11) ☐ The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

Priority under 35 U.S.C. § 119

- 12) ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
- a) ☐ All b) ☐ Some * c) ☐ None of:
1. ☐ Certified copies of the priority documents have been received.
2. ☐ Certified copies of the priority documents have been received in Application No. _____.
3. ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

* See the attached detailed Office action for a list of the certified copies not received.

Attachment(s)

- 1) ☐ Notice of References Cited (PTO-892)
- 2) ☐ Notice of Draftsperson's Patent Drawing Review (PTO-948)
- 3) ☐ Information Disclosure Statement(s) (PTO/SB/08)
Paper No.(s)/Mail Date _____
- 4) ☐ Interview Summary (PTO-413)
Paper No.(s)/Mail Date _____
- 5) ☐ Notice of Informal Patent Application
- 6) ☐ Other: _____

Application/Control Number: 10/501,277
Art Unit: 1795

Page 2

DETAILED ACTION

The text of those sections of Title 35, U.S. Code not included in this action can be found in a prior Office action.

Claim Rejections - 35 USC § 103

Claims 20-54 stand rejected under 35 U.S.C. 103(a) as being unpatentable over Kasaalan in view of Leonard of record for reasons of record.

Response to Arguments

Applicant's arguments filed 1/23/09 have been fully considered but they are not persuasive.

Applicants argue that "With all of the foregoing background information and explanatory disclosure, it is difficult to imagine that Kasaalan would later contradict himself in the same document. Yet in the Office Action, the Examiner relies on Kasaalan's Example 1 (in columns 4 and 5) for the proposition that he discloses a leach process that uses "sulfuric acid with sulfur dioxide bubbled through the tank," from which the Examiner infers that both the sulphur dioxide AND the sulphuric acid are somehow involved in the leaching reaction itself, or that both function as the leaching

agents. This is a misapprehension of Kasaalan's teaching, and it exalts form over substance to insist that in Example 1, Kasaalan is disclosing a *different* function for the sulphur dioxide than that which he discloses over and over again elsewhere, in the more general, prefatory portions of his patent."

The Kasaalan patent discloses the same steps of treating a manganese containing material with an acidic leach solution and passing a volume of sulphur dioxide through the solution to leach the ore (see example 1). The sulfur dioxide is added to the leach solution and forms a leach solution with the sulfuric acid (see col. 3, lines 30-40).

In response to applicant's argument that Kasaalan would use the sulfur dioxide for a different reason, the fact that applicant has recognized another advantage which would flow naturally from following the suggestion of the prior art cannot be the basis for patentability when the differences would otherwise be obvious. See *Ex parte Obiaya*, 227 USPQ 58, 60 (Bd. Pat. App. & Inter. 1985).

Applicants further argue the Kasaalan's process includes further steps that are required to accomplish the desired result, whereas the present invention does not require such steps. The claim language of the instant claims is written in open language and do not exclude further steps.

Applicants further argue that the sulfuric acid is merely present in the solution to lower the pH to 1.5 or below and other acids can be used. This disclosure is matched by the disclosure of Kasaaian, which teaches the use of the sulfuric acid to lower the pH of the solution to around 1.5 and other acids can theoretically be used (see col. 3, lines 17-24). This lower pH would inherently minimize the formation of dithionate ions as noted on page 7, lines 13-19 of the specification.

With respect to the Leonard patent, applicants argue that the patent is only talking about the starting material and even though the patent teaches that these are operating variables.

References are valid for what they convey, explicitly or implicitly, to one skilled in art; that experimentation may not have appeared promising is of no importance; reference may be valid even though it states that its disclosure is not practical. *In re Aller et al*, 105 U.S.P.Q.233 C.C.P.A. (1955).

Consequently, it would have been obvious to a person of ordinary skill in the art at the time the invention was made to modify Kasaaian by the teachings of Leonard.

One having ordinary skill in the art would have been motivated to do this modification, because Leonard discloses that the values of the dithionate present during operation of a manganese leaching process should be maintained or adjusted within the claimed range (col. 4, lines 33-50).

Conclusion

THIS ACTION IS MADE FINAL. Applicant is reminded of the extension of time policy as set forth in 37 CFR 1.136(a).

A shortened statutory period for reply to this final action is set to expire **THREE MONTHS** from the mailing date of this action. In the event a first reply is filed within **TWO MONTHS** of the mailing date of this final action and the advisory action is not mailed until after the end of the **THREE-MONTH** shortened statutory period, then the shortened statutory period will expire on the date the advisory action is mailed, and any extension fee pursuant to 37 CFR 1.136(a) will be calculated from the mailing date of the advisory action. In no event, however, will the statutory period for reply expire later than **SIX MONTHS** from the mailing date of this final action.

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Arun S. Phasge whose telephone number is (571) 272-1345. The examiner can normally be reached on **MONDAY-THURSDAY, 7:30-6:00.**

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Nam X. Nguyen can be reached on (571) 272-1342. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Application/Control Number: 10/501,277
Art Unit: 1795

Page 6

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/Arun S. Phasge/
Primary Examiner, Art Unit 1795

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AMENDMENTS TO THE CLAIMS:

29. (Currently Amended) A process for the hydrometallurgical processing of manganese containing materials, the process characterised by the combination of a manganese dioxide containing feedstock and an acidic solution to form an acidic a leach solution to be leached, and passing a volume of sulphur dioxide gas through that leach solution as the leaching agent, whereby the levels of dithionate ion generated in the leach solution are less than about 5g/l.

30. (Currently Amended) A process according to claim 429, wherein the levels of dithionate ion generated in the leach solution are less than about 1g/l.

31. (Currently Amended) A process according to claim 429, wherein the pH of the leach solution is maintained at less than about 1.5.

32. (Currently Amended) A process according to claim 429, wherein the leach solution comprises a slurry of manganese dioxide containing material at a slurry density of less than about 10%w/v, less than about 120g/l manganese sulphate, a temperature of greater than about 95°C; and at a pH of less than about 1.5.

33. (Currently Amended) A process according to claim 429, wherein the

Docket No.: 4046/022

manganese dioxide containing feedstock contains less than about 40% manganese.

34. (Currently Amended) A process according to claim 129, wherein the leach solution has an initial soluble iron concentration of greater than 4g/l.

35. (Currently Amended) A process according to claim 634, wherein the iron is in the form of ferric sulphate ($\text{Fe}_2(\text{SO}_4)_3$).

36. (Currently Amended) A process according to claim 429, wherein the ferrous concentration is maintained at a level below about 0.5g/l by providing an excess or residual amount of manganese dioxide in the slurry.

37. (Currently Amended) A process according to claim 429, wherein the ratio of ferric to ferrous is monitored at least at intervals throughout the leach to ensure an oxidation reduction potential (ORP) of 550mV, or above (vs Ag/AgCl reference electrode).

38. (Currently Amended) A process according to claim 429, wherein the sulphur dioxide gas is preferably passed through the leach solution over a period of at least 10 hours whereby up to about 95% of manganese dioxide is dissolved.

39. (Currently Amended) A process according to claim ~~429~~, wherein the leach is conducted over a period of between about 10 to 15 hours.

40. (Currently Amended) A process according to claim ~~429~~⁴²⁸, wherein once a stoichiometric amount of sulphur dioxide has been added to the leach solution to achieve a 95% dissolution of the manganese dioxide present, the reaction is halted.

41. (Currently Amended) A process for the production of electrolytic manganese dioxide, the process characterised by a leach of a manganese dioxide containing feedstock ~~in acidic solution, through in~~ which a volume of sulphur dioxide gas ~~as the leaching agent~~ is passed ~~through an acidic solution containing manganese dioxide, and in which the~~ dithionate ion levels ~~are in said solution being~~ maintained at less than about 5g/l, the resulting leach solution being processed to provide an appropriate electrolyte that is passed to an electrowinning stage during which electrolytic manganese dioxide is deposited.

42. (Currently Amended) A process according to claim ~~1341~~1341, wherein the levels of dithionate ion generated in the leach solution are less than about 1g/l.

43. (Currently Amended) A process according to claim ~~43~~41, wherein the pH of the leach solution is maintained at less than about 1.5.

44. (Currently Amended) A process according to claim ~~43~~41, wherein the leach solution comprises a slurry of manganese dioxide containing material at a slurry density of less than about 10%w/v, less than about 120g/l manganese sulphate, a temperature of greater than about 95°C, and at a pH of less than about 1.5.

45. (Currently Amended) A process according to claim ~~43~~41, wherein the manganese dioxide containing feedstock contains less than 40% manganese.

46. (Currently Amended) A process according to claim ~~1341~~ 1341, wherein the leach solution has an initial soluble iron concentration of greater than 4g/l.

47. (Currently Amended) A process according to claim ~~1846~~, wherein the iron is in the form of ferric sulphate ($\text{Fe}_2(\text{SO}_4)_3$).

48. (Currently Amended) A process according to claim ~~1341~~, wherein the ferrous concentration is maintained at a level below about 0.5g/l by providing an excess or residual amount of manganese dioxide in the slurry.

49. (Currently Amended) A process according to claim ~~43~~41, wherein the ratio of ferric to ferrous is monitored throughout the leach to ensure an oxidation reduction potential (ORP) of 550mV, or above (vs Ag/AgCl reference electrode).

50. (Currently Amended) A process according to claim ~~1341~~ 1341, wherein the sulphur dioxide gas is passed through the leach solution over a period of at least 10 hours whereby up to about 95% of manganese dioxide is dissolved.

51. (Currently Amended) A process according to claim ~~41~~41, wherein the leach is conducted over a period of between about 10 to 15 hours.

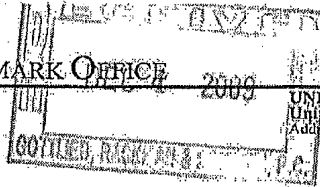
52: (Currently Amended) A process according to claim ~~1341~~, wherein once a stoichiometric amount of sulphur dioxide has been added to the leach solution to achieve a 95% dissolution of the manganese dioxide present, the reaction is halted.

53. (Currently Amended) A process according to claim ~~43~~41, wherein the acidic solution used in the leach is at least in part comprised of return or spent sulphuric acid solution from the electrowinning stage.

54. (Currently Amended) A process according to claim ~~1341~~, wherein additional acid is added to the leach to ensure the pH remains less than about 1.5.



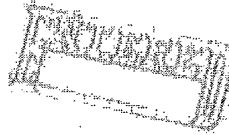
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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/501,277	09/03/2004	Christopher Brett Ward	4046-022	5676

22440: 7590 12/01/2009
GOTTLIEB RACKMAN & REISMAN PC
270 MADISON AVENUE
8TH FLOOR
NEW YORK, NY 10016-0601



EXAMINER

PHASGE, ARUN S

ART UNIT	PAPER NUMBER
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1795

MAIL DATE	DELIVERY MODE
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12/01/2009

PAPER

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The time period for reply, if any, is set in the attached communication.

Office Action Summary

Application No.

10/501,277

Applicant(s)

WARD, CHRISTOPHER BRETT

Examiner

Arun S. Phasge

Art Unit

1795

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) OR THIRTY (30) DAYS; WHICHEVER IS LONGER, FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED. (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

Status

- 1) ☒ Responsive to communication(s) filed on 14 September 2009.
- 2a) ☐ This action is FINAL. 2b) ☒ This action is non-final.
- 3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

Disposition of Claims

- 4) ☒ Claim(s) 29-54 is/are pending in the application.
- 4a) Of the above claim(s) _____ is/are withdrawn from consideration.
- 5) ☐ Claim(s) _____ is/are allowed.
- 6) ☒ Claim(s) 29-54 is/are rejected.
- 7) ☐ Claim(s) _____ is/are objected to.
- 8) ☐ Claim(s) _____ are subject to restriction and/or election requirement.

Application Papers

- 9) ☐ The specification is objected to by the Examiner.
- 10) ☐ The drawing(s) filed on _____ is/are: a) ☐ accepted or b) ☐ objected to by the Examiner.
Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).
Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).
- 11) ☐ The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

Priority under 35 U.S.C. § 119

- 12) ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
- a) ☐ All b) ☐ Some * c) ☐ None of:
- ☐ Certified copies of the priority documents have been received.
 - ☐ Certified copies of the priority documents have been received in Application No. _____.
 - ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

* See the attached detailed Office action for a list of the certified copies not received.

Attachment(s)

- ☐ Notice of References Cited (PTO-892)
- ☐ Notice of Draftsperson's Patent Drawing Review (PTO-948)
- ☐ Information Disclosure Statement(s) (PTO/SB/08)
Paper No(s)/Mail Date _____
- ☐ Interview Summary (PTO-413)
Paper No(s)/Mail Date _____
- ☐ Notice of Informal Patent Application
- ☐ Other: _____

DETAILED ACTION

Claim Rejections - 35 USC § 112

Claims 29-54 rejected under 35 U.S.C. 112, first paragraph, as failing to comply with the written description requirement. The claim(s) contains subject matter which was not described in the specification in such a way as to reasonably convey to one skilled in the relevant art that the inventor(s), at the time the application was filed, had possession of the claimed invention.

The portion of the specification pointed out to provide support for the new limitation is not adequate for the limitations presently recited in the claims and presently added to the specification.

Claim Rejections - 35 USC § 103

The text of those sections of Title 35, U.S. Code not included in this action can be found in a prior Office action.

Claims 29-54 are rejected under 35 U.S.C. 103(a) as being unpatentable over Kaasiah in view of Leonard of record for reasons of record.

The Kaasian patent further discloses the use of different sources of mananomanganic oxide, including sintered manganese ore or natural manganese ore (col. 2, lines 44-46).

Consequently, while the preferred embodiment uses a sintered ore, the patent teaches the use of natural manganese ore as an equivalent to the sintered ore. Accordingly, even if the added limitations are considered, the Kaasian patent teaches the use of un-sintered or natural ore.

Response to Arguments

Applicant's arguments with respect to claims have been considered but are moot in view of the new ground(s) of rejection.

Conclusion

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Arun S. Phasge whose telephone number is (571) 272-1345. The examiner can normally be reached on MONDAY-THURSDAY, 7:30-6:00.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Nam X. Nguyen can be reached on (571) 272-1342. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

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Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

/Arun S. Phasge/
Primary Examiner, Art Unit 1795

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